



Docket No.: 50195-261

PATENT**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Inventor Application of

Hideaki ONO, et al.

Serial No.: 09/893,892

Filed: June 29, 2001

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: Confirmation Number: 4949

: Group Art Unit: 1742

: Examiner: J. Sheehan

For: **EXCHANGE SPRING MAGNET POWDER AND A METHOD OF PRODUCING THE SAME****Declaration Under 37 C.F.R. § 1.132**

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Dr. Munekatsu Shimada, declare as follows:

1. I received a doctorate of Engineering from the University of Tokyo Department of Applied Physics.
2. My field of specialty is magnetic materials.
3. I am employed as a Senior Engineer at Technology Research Laboratory No. 1, Nissan Research Center. I have been employed by Nissan Motor Co., Ltd. for 23 years.
4. I am a coinventor of U.S. Patent Application Serial No. 09/893,892, EXCHANGE SPRING MAGNET POWDER AND A METHOD OF PRODUCING THE SAME, filed June 29, 2001.

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5. I have read and am familiar with the disclosure of the above-captioned patent application. I have read and am familiar with the disclosure of Nomura et al., U. S. Patent No. 6,261,385 (the '385 patent).

6. At my direction and under my supervision, magnet powder was produced by the methods described in both the '385 patent and according to the methods of the instant invention, by preparing a starting alloy having a chemical composition described in Example 3 of the '385 patent.

7. Nanocomposite magnet powder was formed by:

(Step 1) Preparing a starting alloy having a chemical composition corresponding to the formula $\text{Nd}_9\text{Fe}_{86}\text{B}_5$ (Example 3 of the '385 patent).

(Step 2) Melting the starting alloy by high-frequency induced heating, under an atmosphere of argon

(Step 3) Quenching the melted alloy obtained by Step 2 by using the liquid quenching method with a single roller having the rolling velocity of about 24 m/sec. under an atmosphere of argon to obtain a 100 % amorphous thin ribbon.

(Step 4) Pulverizing the alloy quenched thin ribbon obtained by Step 3 to a powder with a particle diameter below about 350 μm under an atmosphere of argon.

(Step 5) Placing the powder obtained in Step 4 in a hermetically-closed, soft iron, cylindrical container, which has an inside diameter of about 14mm and height of about 30 mm, heating the alloy powder at about 900 °C by high-frequency induced heating, and compressing about 10 g of the pulverized powder, in the direction of the arrow in Fig 1 by using crank-type press machine with a distortion strain rate of about 150/sec and a compression ratio within the range from 90 % to 95 %.

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8. Fig. 1 is a photomicrograph showing the microstructure of the nanocomposite magnet powder produced by using the method of paragraph 7. The photomicrograph was taken by a HITACHI H-9000 High Resolution Transmission Electron Microscope (TEM) having an ability to accelerate with the maximum voltage 300 kV. The thickness of the sample reduced by Focused Ion Beam (FIB) machining was about 100 nm. As shown in Fig. 1, the nanocomposite magnet powder produced by this method has crystal particles with a diameter of about 150 nm.

9. Exchange spring magnet powder was formed by:

(Step 1) Preparing a starting alloy having a chemical composition corresponding to the formula $\text{Nd}_9\text{Fe}_{86}\text{B}_5$ (Example 3 of the '385 patent).

(Step 2) Melting the starting alloy by high-frequency induced heating, under an atmosphere of argon.

(Step 3) Quenching the melted alloy obtained by Step 2 by using the liquid quenching method with a single roller having the rolling velocity of about 10 m/sec., under an atmosphere of argon, to provide a 30 % amorphous thin ribbon.

(Step 4) Pulverizing the alloy quenched thin ribbon obtained by Step 3 to a powder with a powder size below 500 μm under an atmosphere of argon.

(Step 5) Amorphizing 20 g of the alloy powder, obtained by Step 4, by using the ball mill method as follows:

mixing the alloy powder in a stainless steel ball mill pot together with 200 g of stainless steel balls and cyclohexane as a solvent, sealing the pot under an atmosphere of argon, and ball milling for 16 hours.

(Step 6) Crystallizing the amorphised alloy by heat treating at 600 °C for 10 min. under vacuum.

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10. Fig. 2 is a photomicrograph showing the microstructure of the nanocomposite magnet powder produced by using the method of paragraph 9. The photomicrograph was taken by a HITACHI H-9000 High Resolution Transmission Electron Microscope (TEM) having an ability to accelerate with the maximum voltage 300 kV. The thickness of the sample reduced by Focused Ion Beam (FIB) machining was about 100 nm. As shown in Fig. 2, the nanocomposite magnet powder produced by this method has crystal particles with a diameter of about 50 nm.

11. The steps listed in paragraph 7 correspond to the method of forming a nanocomposite magnet powder in the '385 patent. The steps described in paragraph 9, are within the scope of claim 14 of the instant patent application.

12. The exchange spring magnet powder produced by the method described in paragraph 9 has a smaller crystal particle diameter, about 50 nm, than the nanocomposite magnet produced by the method described in paragraph 7, about 150 nm.

13. The amorphous content of the quenched thin ribbon obtained in Step 3 of the method paragraph 7, 100 % amorphous, is greater than the amorphous content of the quenched thin ribbon obtained in Step 3 of the method of paragraph 9, 30 % amorphous.

14. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements are made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

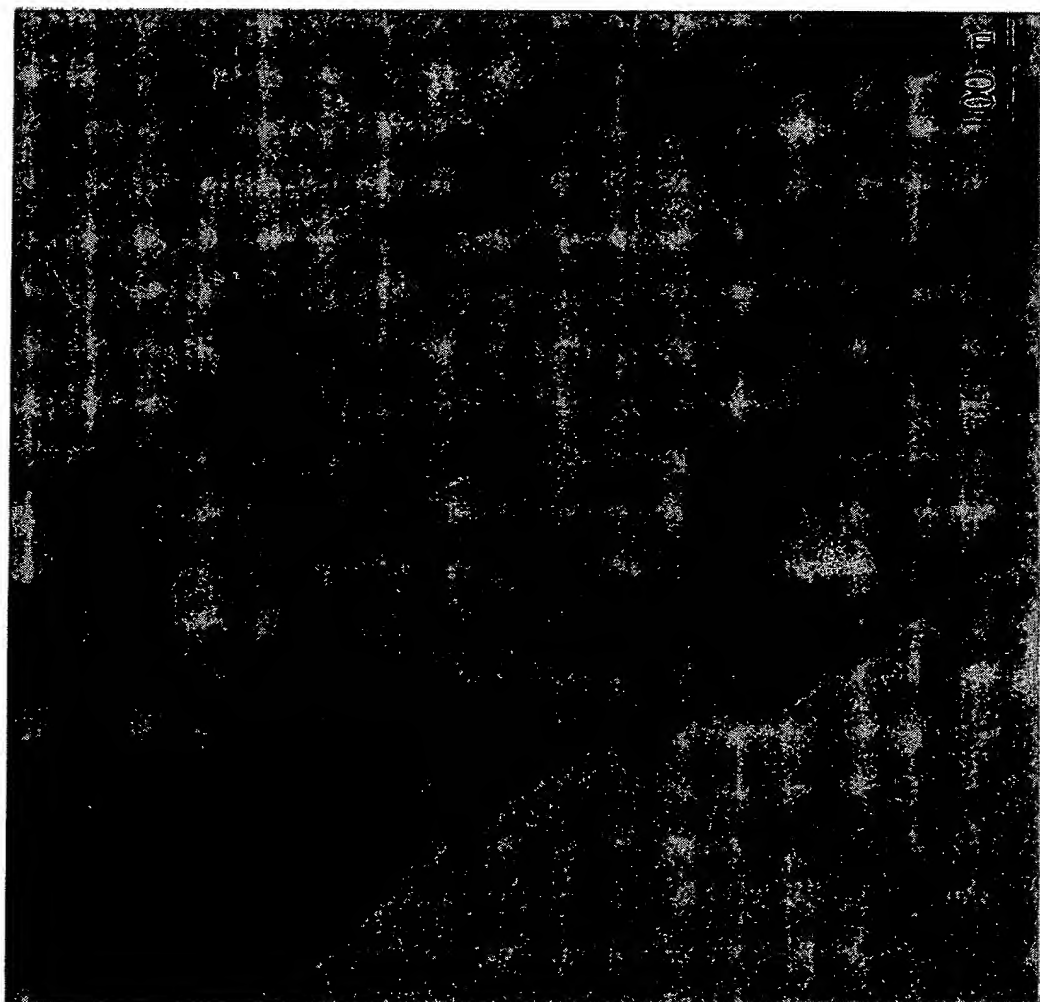
10/28/2004
Date

Munekatsu Shimada
Dr. Munekatsu Shimada



100nm

添付図1
Fig. 1



添付図 2

Fig. 2

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